भारतीय मानक

Indian Standard

IS 9909 : 2023

इलेक्ट्रोलेस चढ़ाना के लिए सक्सिनिक एसिड के लिए विनिर्देश

(पहला पुनरीक्षण)

Specification for Succinic Acid for Electroless Plating

(First Revision)

ICS 34.040.30

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भारतीय मानक ब्यूरो
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June 2023 **Price Group 6**

Electroplating Chemicals and Photographic Materials Sectional Committee, CHD 05

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Electroplating Chemicals and Photographic Materials Sectional Committee had been approved by the Chemical Division Council.

Succinic acid and succinates are used as 'buffering agents' for controlling *p*H and as 'exaltants' for increasing deposition rates in acidic electroless plating baths using sodium hypophosphite as reducing agent. They are also used as 'complexing agents' to prevent precipitation of nickel hydroxide in alkaline electroless plating baths using sodium borohydride as reducing agent.

This standard was first published in 1981 and is being revised considering advancement in technology for testing of the product. In this revision following changes have been done:

a) Incorporation of the requirement of arsenic and its test method; and
b) Incorporation of alternate instrumental test method (AAS) for Pb, Fe and As.

The composition of the committee responsible for formulation of this standard is listed in Annex D.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 2022 'Rules for rounding off numerical values (*second revision*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

SPECIFICATION FOR SUCCINIC ACID FOR ELECTROLESS PLATING

(First Revision)

1 SCOPE

This standard prescribes requirements and methods of sampling and test for succinic acid for electroless plating.

2 REFERENCES

The standards listed in Annex A contains provisions which through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated in Annex A.

3 REQUIREMENTS

3.1 Description

Succinic acid shall be in the form of colourless crystals readily soluble in water, free from foreign matter and visible impurities and shall correspond essentially to the formula $(CH_2CO_2H)_2$.

3.2 The material shall also comply with the requirements prescribed in Table 1 when tested according to methods prescribed in Annex B. Reference to the relevant clauses of Annex B is given in co1 (4) of Table 1.

Table 1 Requirements for Succinic Acid for Electroless Plating
(*Clause* 3.2)

Sl No. (1)	Characteristic (2)	Requirement (3)	Method of Test, (Ref to Clause No. in Annex B) (4)
i)	Melting range, °C	l81 - 185	**B-2**
ii)	Matter insoluble in water, percent by mass, *Max*	0.01	**B-3**
iii)	Ignition residue, percent by mass, *Max*	0.1	**B-4**
iv)	Chloride (as Cl), percent by mass, *Max*	0.1	**B-5**
v)	Sulphate (as SO_4), percent by mass, *Max*	0.05	**B-6**
vi)	Heavy metals (as Pb), percent by mass, *Max*	0.002	**B-7**
vii)	Iron (as Fe), percent by mass, *Max*	0.002	**B-8**
viii)	Arsenic (as As), percent by mass, *Max*	2.0	**B-9**
viii)	Assay (as $(CH_2CO_2H)_2$), percent by mass, *Min*	99.0	**B-10**

4 PACKING AND MARKING

4.1 Packing

The material shall be packed in air-tight containers preferably with replaceable closure. The containers shall be marked with the following:

a) Name and net mass of the material;

b) Name of the manufacturer or his trade-mark, if any; and

c) Date and batch number of manufacture to enable the material to be traced from records.

4.2 BIS Certification Marking

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act*, 2016 and the Rules and Regulations framed thereunder, and the products may be marked with the Standard Mark.

5 SAMPLING

The method of preparing representative samples of the material and criteria for its conformity with this standard shall be as prescribed in Annex C.

ANNEX A

(*Clause* 2)

LIST OF REFERRED INDIAN STANDARDS

IS No.	*Title*
IS 264 : 2005	Nitric acid — Specification (*third revision*)
IS 265 : 2021	Hydrochloric acid — Specification (*fifth revision*)
IS 266 : 1993	Sulphuric acid — Specification (*third revision*)
IS 1070 : 1992	Reagent grade water — Specification (*third revision*)
IS 2088 : 1983	Methods for determination of arsenic (*second revision*)
IS 4825 : 1982	Specification for liquid-in-glass solid-stem reference thermometers (*first revision*)
IS 4905 : 2015	Random sampling and randomization procedures (*first revision*)
IS 5762 : 1970	Methods for determination of melting point and melting range
IS 11124 : 1984	Method for atomic absorption spectrophotometric determination of arsenic
IS 12074 : 1987	Method for determination of lead by atomic absorption spectrophotometr
IS 13320 : 1992	Determination of iron by atomic absorption spectrometry — Test method

ANNEX B

(*Clause* 3.2)

METHODS OF TEST FOR SUCCINIC ACID FOR ELECTROLESS PLATING

B-l QUALITY OF REAGENTS

B-l.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be used in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

B-2 DETERMINATION OF MELTING RANGE

B-2.1 General

The melting range is determined by the capillary tube method.

B-2.2 Procedure

Determine the melting range of the material as prescribed in IS 5762 (*see* thermometer corresponding to Schedule Mark 25 of IS 4825).

B-3 TEST FOR MATTER INSOLUBLE IN WATER

B-3.1 Procedure

Weigh accurately 10 g of the material and dissolve in 150 ml of hot water. Heat on a steam bath for 30 min with occasional stirring. Filter through a sintered glass crucible (G. No. 4), wash the residue with hot water and dry at 105 °C ± 2 °C. Cool in a desiccator and weigh. Reserve the filtrate for further tests.

B-3.2 Calculation

Matter insoluble in water, percent by mass

$$= \frac{M_1 \times 100}{M} \qquad \text{….(1)}$$

where

M_1 = mass, in g, of the residue; and
M = mass, in g, of the material taken for the test.

B-4 TEST FOR IGNITXON RESIDUE

B-4.1 Procedure

Weigh accurately about 4 g of the material and ignite in a porcelain crucible till constant mass is achieved.

B-4.2 Calculation

Ignition residue, percent by mass

$$= \frac{M_1 \times 100}{M} \qquad \text{…..(2)}$$

where

M_1 = mass, in g, of the residue; and

M = mass, in g, of the material taken for the test.

B-5 TEST FOR CHLORIDE

B-5.1 Apparatus

B-5.1.1 *Nessler Cylinders,* Two of 50 ml capacity each.

B-5.2 Reagents

B-5.2.1 *Concentrated Nitric Acid, see* IS 264.

B-5.2.2 *Silver Nitrate Solution,* 1.7 percent (*m/v)* in water.

B-5.2.3 *Standard Chloride Solution*

Dissolve 0.165 g of sodium chloride (NaCl) in water, and dilute with water up to the mark in a l 000 ml volumetric flask. Dilute 10 ml of this solution in l00 ml volumetric flask. (1 ml solution = 0.01 mg of Cl^-).

B-5.3 Preparation of the Test Solution

Transfer the cooled filtrate and washings reserved in **B-3.1** to a 250 ml volumetric flask and dilute with water up to the mark.

B-5.4 Procedure

Transfer 25 ml aliquot of the prepared sample solution (**B-5.3**) to a 250 ml volumetric flask and dilute to 250 ml. Take 25 ml of the diluted solution in a 50 ml Nessler cylinder, add 1 ml of concentrated nitric acid followed by 1 ml of silver nitrate solution. Make up to the mark with water, mix and allow the turbidity to develop. Carry out a control test in another Nessler cylinder using 1 ml of standard chloride solution and the same quantities of other reagents in the same total volume as with the material and compare the turbidity produced in two cylinders against a black background.

B-5.4.1 The material shall be taken as having satisfied the requirement of the test if turbidity produced with the material is not greater than that produced in the control test.

B-6 TEST FOR SULPHATE

B-6.1 General

Turbidity produced by the addition of barium chloride solution containing the material is compared with that produced in the blank containing standard sulphate solution.

B-6.2 Apparatus

B-6.2.1 *Nessler Cylinders,* Two of 100 ml capacity each.

B-6.3 Reagents

B-6.3.1 *Concentrated Hydrochloric Acid, see* IS 265.

B-6.3.2 *Barium Chloride Solution*

Dissolve 12 g of barium chloride in 100 ml of water.

B-6.3.3 *Standard Sulphate Solution*

Weigh accurately 0.181 5 g of potassium sulphate (K_2SO_4) and dissolve in water in a 100 ml volumetric flask. Make up the volume with water and mix. (1 ml solution = 0.1 mg SO_4^{2-})

B-6.4 Procedure

Transfer 20 ml of aliquot of the sample solution prepared in **B-5.3** to a l00 ml Nessler cylinder and add 1.0 ml of concentrated hydrochloric acid followed by 5 ml of barium chloride solution. Make up the volume with water and mix. Carry out a control test in another Nessler cylinder using 4 ml of standard sulphate solution and the same quantities of other reagents in the same total volume as with the material and compare the turbidity produced in the two cylinders against a black background.

B-6.4.1 The material shall be taken as having satisfied the requirement of the test if turbidity produced with the material is not greater than that produced in the control test.

B-7 TEST FOR HEAVY METALS

B-7.1 General

Two methods for the determination of heavy metals are prescribed. Either of these may be used for general routine purposes, but in case of a dispute Method A shall be used as referee method.

B-7.2 Method A

B-7.2.1 *Apparatus*

B-7.2.1.1 *Nessler cylinders,* Two of 50 ml capacity each.

B-7.2.2 *Reagents*

B-7.2.2.1 *Ammonium hydroxide*

B-7.2.2.2 *Dilute hydrochloric acid*, approximately 1 N.

B-7.2.2.3 *Hydrogen sulphide solution*

A freshly prepared, saturated aqueous solution of hydrogen sulphide gas.

B-7.2.2.4 *Standard lead solution*

Dissolve 0.160 g of lead nitrate [$Pb(NO_3)_2$] in 100 ml of water containing 1 ml of concentrated nitric acid and dilute with water to 1 000 ml in a 1 000 ml volumetric flask. Dilute 10 ml of this solution to 100 ml with water in a l00 ml volumetric flask. (1 ml solution = 0.01 mg of Pb)

B-7.2.3 *Procedure*

Weigh accurately 5.00 g of the material and dissolve it in 40 ml of hot water and neutralize with ammonium hydroxide to litmus paper. Cool, add 5 ml of dilute hydrochloric acid and dilute to 50 ml with water in a 50 ml volumetric flask. Transfer 30 ml of this solution to Nessler cylinder A and 10 ml to Nessler cylinder B. Add 4 ml of standard lead solution to cylinder B.

Then add 10 ml of hydrogen sulphide solution to each of the cylinders, dilute to the mark with water and mix.

B-7.2.3.1 The material shall be taken as having satisfied the requirement of the test if the darkness produced in cylinder A is not greater than that produced in cylinder B.

B-7.3 Method B

Determine lead content by atomic absorption spectrophotometer in accordance with method given in IS 12074.

B-8 TEST FOR IRON

B-8.1 General

Two methods for the determination of iron are prescribed. Either of these may be used for general routine purposes, but in case of a dispute method A shall be used as referee method.

B-8.2 Method A

B-8.2.1 *Apparatus*

B-8.2.1.1 *Nessler cylinders,* Two of 100 ml capacity each.

B-8.2.2 *Reagents*

B-8.2.2.1 *Concentrated sulphuric acid, see* IS 266.

B-8.2.2.2 *Ammonium persulphate*

B-8.2.2.3 *Butanolic potassium thiocyanate*

Dissolve 10 g of potassium thiocyanate in 10 ml of water. Warm the solution to about 25 °C to 30 °C. Dilute with n-butanol to 100 ml and shake vigorously.

B-8.2.2.4 *Standard iron solution*

Dissolve 0.702 g of ferrous ammonium sulphate [$FeSO_4(NH_4)_2SO_4.6H_2O$] in water containing 10 ml of concentrated sulphuric acid and dilute to 100 ml with water in a 100 ml volumetric flask. Dilute 10 ml of this solution to 100 ml with water in a 100 ml volumetric flask. (1 ml solution = 0.01 mg of Fe).

B-8.2.3 *Procedure*

Weigh accurately l.00 g of the material and dissolve in 40 ml of water in a Nessler cylinder. Add 2 ml of concentrated sulphuric acid followed by 30 mg of ammonium persulphate and 15 ml butanolic potassium thiocyanate. Shake well and allow the layers to separate. Carry out a control test in another Nessler cylinder using 2 ml of standard iron solution and the same quantities of other reagents in the same total volume as with the material.

B-8.2.3.1 The material shall be taken as having satisfied the requirement of the test if the intensity of the red colour in the butanol layer, produced with the material is not greater than that produced in the control test.

B-8.3 Method B

Determine iron content by Atomic Absorption Spectrophotometer (AAS) in accordance with method given in IS 13320.

B-9 TEST FOR ARSENIC

B-9.1 General

Two methods for the determination of arsenic are prescribed. Either of these may be used for general routine purposes, but in case of a dispute method A shall be used as referee method.

B-9.2 Method A

The stain produced by arsine or mercuric bromide paper is compared with a standard stain.

B-9.2.1 *Reagents*

B-9.2.1.1 *Concentrated hydrochloric acid, see* IS 265.

B-9.2.1.2 *Hydrazine sulphate*

B-9.2.1.3 *Sodium bromide*

B-9.2.2 *Procedure*

B-9.2.2.1 *For technical grade*

Preparation of test solution

Weigh 1.0 g of the material and transfer to a small distillation flask. Add 10 ml of water, 15 ml of concentrated hydrochloric acid, 0.25 g of hydrazine sulphate and 0.25 g of sodium bromide. Connect the flask to a condenser and distil the contents till 20 ml of the distillate are collected. Use this distillate for carrying out the test for arsenic, as described in IS 2088 using for comparison a stain obtained with 0.015 mg of arsenic trioxide.

B-9.2.2.2 *For analytical reagent grade*

Weigh 3.0 g of the material and carry out the test for arsenic by the modified Gutzeit method as prescribed in **5.1** or spectrophotometric method as prescribed in **5.2** of IS 2088 using for comparison in **5.1** a stain obtained with 0.003 mg of arsenic trioxide.

B-9.3 Method B

Determine arsenic content by atomic absorption spectrophotometer (AAS) in accordance with method given in IS 11124.

B-10 TEST FOR SUCCINIC ACID CONTENT

B-10.1 Reagents

B-10.1.1 *Standard Sodium Hydroxide Solution* — approximately 0.1 N.

B-10.1.2 *Phenolphthalein Indicator Solution*

Dissolve 0.1 g of phenolphthalein in 100 ml of 60 percent (*v*/*v*) rectified spirit.

B-10.2 Procedure

Dry about 0.5 g of the material over concentrated sulphuric acid overnight. Then weigh accurately about 0.3 g of the material and dissolve in 25 ml of water. Add 2 drops of phenolphthalein indicator solution and titrate with standard sodium hydroxide solution.

B-10.3 Calculation

Succinic acid, content, percent by mass

$$= \frac{5.905 \times V \times \mathrm{N}}{M} \qquad \ldots (3)$$

where

V = volume, in ml, of standard sodium hydroxide solution consumed;

N = normality of standard sodium hydroxide solution; and

M = mass, in g, of the material taken for the test.

ANNEX C

(*Clause* 5.1)

SAMPLING OF SUCCINIC ACID FOR ELECTROLESS PLATING

C-1 GENERAL REQUIREMENTS OF SAMPLING

C-1.1 In drawing, preparing, storing and handling samples, the following precautions and directions shall be observed.

C-1.2 Samples shall be taken in a protected place not exposed to damp air, dust or soot.

C-l.3 The sampling instrument shall be clean and dry.

C-l.4 Precautions shall be taken to protect, the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

C-1.5 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

C-l.6 The samples shall be placed in suitable, clean, dry and air-tight containers on which the material has no action.

C-l.7 The sample containers shall be of such a size that they are almost completely filled by the sample.

C-l.8 Each sample container shall be sealed air-tight after filling and marked with full details of sampling, the date of sampling, year of manufacture and other important particulars of consignment.

C-2 SCALE OF SAMPLING

C-2.1 Lot

All the containers in a single consignment of the material drawn from a single batch of manufacture shall constitute the lot. If a consignment is declared to consist of different batches of manufacture, the batches shall be marked separately and the groups of containers in in each batch shall constitute separate lots.

C-2.2 For ascertaining the conformity of the material in any lot to the requirements of this specification, samples shall be tested for each lot separately.

C-2.3 The number of containers to be selected from peach lot shall depend on the size of the lot and shall be in accordance with co1 (1) and (2) of Table 2.

Table 2 Number of Containers to be Selected for Sampling

(*Clause* C-2.3)

Sl No. (1)	Lot Size (2)	Number of Containers to be Selected (3)
i)	up to 50	3
ii)	51 to 100	4
iii)	101 to 150	5
iv)	151 to 300	6
v)	301 to 500	7
vi)	501 and above	8

C-2.3.1 These containers shall be selected at random. In order to ensure the randomness of selection, procedures given in IS 4905 may be followed.

C-3 TEST SAMPLE AND REFEREE SAMPLE

C-3.1 From each of the containers selected as in **C-2.3**, draw with an appropriate sampling instrument small portions of the material from different parts of the containers. The total quantity so drawn from each of the containers shall be approximately equal to thrice the quantity required for testing purposes.

C-3.2 Mix thoroughly all the portions of the material drawn from the same container to give a representative sample for the container.

C-3.3 From the samples **(C-3.2)** representing different containers selected in **C-2.3**, a small but equal quantity of material shall be taken and thoroughly mixed to form a composite sample. The composite sample so obtained shall be divided into three equal parts, one for the purchaser, another for the supplier and the third for the referee.

C-3.4 The remaining portion of the material in the samples **(C-3.2)** from different containers shall be divided into three equal parts, each forming an individual sample. One set of individual samples representing the containers selected shall be for the purchaser, another for the supplier and the third for the referee.

C-3.5 All the individual and composite samples shall be transferred to separate containers. These containers shall then be sealed air-tight with stoppers and labelled with full identification particulars given in **C-1.8**.

C-3.6 The referee samples consisting of a composite sample and a set of individual samples shall bear the seals of both the purchaser and the supplier and shall be kept at a place agreed to between the two. This shall be used in case of any dispute between the two.

C-4 NUMBER OF TESTS

C-4.1 Tests for the determination of succinic acid content shall be carried out on each of the individual samples.

C-4.2 Tests for the remaining characteristics shall be carried out on the composite sample.

C-5 CRITERIA FOR CONFORMITY

C-5.1 For Individual Samples

For succinic acid content, the mean and range of test results shall be computed as follows:

Mean ($\bar{X}$) = Sum of individual test results number of tests; and

Range ($\bar{R}$) = Difference between the maximum and the minimum values of the test results.

C-5.1.1 The lot shall be declared to have satisfied the requirement for succinic acid content if $\bar{X} - 0.6\,R \geq 99.0$.

C-5.2 For Composite Sample

For declaring the conformity of the lot to the requirements of all other characteristics, the test results on the composite sample shall meet the corresponding specified requirements.

ANNEX D

(*Foreword*)

COMMITTEE COMPOSITION

Electroplating Chemicals and Photographic Materials Sectional Committee, CHD 05

Organization	*Representative(s)*
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